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Estimation of Microcrystalline Wax in Beeswax

The detection and determination of mineral waxes in beeswax has been a problem for many years (1). Analytical constants for beeswax show considerable natural variation, so that although the presence of large amounts of non-reactive petroleum wax can easily be shown, use of these constants (saponification, acid, ester, and ratio numbers, and melting point) will not reliably indicate less than about 10% of such material in beeswax. The saponification cloud test has been proposed as a more sensitive qualitative test (2).

Examples from the literature of the ranges of values for these constants for yellow beeswax are given in Table 1. Their extent indicates their relative insensitivity for detecting admixture of hydrocarbons with beeswax. The hydrocarbon content is a more direct measure and has been proposed several times (5). Hydrocarbons are a natural constituent of beeswax; here again the natural variability must be established before reliable analyses can be made. Table 2 shows hydrocarbon values for beeswax from the literature. Of these, only Kebler and Bruening analyzed United States beeswax. Further data on the normal range of hydrocarbon content for domestic beeswax are needed.

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We have determined the hydrocarbon content of 59 samples of crude yellow beeswax of known source from 20 states. In order to establish the authenticity of these samples, other analytical "constants" were also determined. These were melting point, saponification number, acid number, ester number, ratio number, and color. In the course of this work the freezing point of the beeswax hydrocarbon was also determined and found to be relatively constant. This value is much more sensitive to the addition of microcrystalline wax than is the melting point of the original beeswax.

MATERIALS AND METHODS

Beeswax Samples

Samples of ½ to 2 pounds of beeswax were collected from producers by the Bee Industries Association. Cappings wax and old comb

Table 2. Hydrocarbon content of beeswax

Value, %	Reference
10.4 –13.0	Leys (6)
12.7 –13.0	Buisine (7)
12.5 –14.0	Kebler (8)
12.5 –14.5	Buisine (9)
12.8 –17.3	Ahrens & Hett (10)
14.50–16.30	Vizern & Guillot (11)
13.6 ±0.48	Curylo & Zalewski (12)
14.93	Bruening (13)
12.28–17.09	This paper

Table 1. Standards for yellow beeswax

Pharmacopoeia[a]	Acid No.	Sapon. No.	Ester No.	M.P.	Ratio No.
U.S. (XV)	18–24		72–77	62–65°C	
German (DAB 6)	16.8–22.1		65.9–82.1	62–66.5	3.0–4.3
French	16.8–22.4	92–102	72–80	62–66	
British	17–23		70–80	62–64	3.3–4.2
U.S.S.R.	17–20.5		66–76	63–65	3.42–3.9
Govt. Spec.					
U.S.[b]	16.5–21.0	86–96		60.5–64.0	3.5–4.3
New Zealand[c]	17–21	87–103	70–80	62–64	3.3–4.2
TGA[d]	17–24	89–103	72–79	62–65	3.3–4.0

[a] From (3). [b] C.B.–191a (4). [c] N.Z.S.S. 743, 1950. [d] Toilet Goods Association, 1959 (in part).

Table 3. Analytical values for American yellow beeswax

Value	This Paper				Bisson, et al. (24)[a]			
	Mean	Range	s	C_v	Mean	Range	s	C_v
M.P.	63.56°	62.68–64.42°	0.35°	0.55%	64.1°	63.1 –65.0°	0.42°	0.65%
Acid No.	18.33	16.68–20.12	0.64	3.49%	18.6	16.8 –20.4	0.86	4.62%
Sapon. No.	90.94	88.62–94.39	1.35	1.38%	93.9	89.3 –99.3	1.84	1.96%
Ester No.	72.61	70.82–75.32	1.10	1.51%	75.3	71.1 –78.9	1.44	1.91%
Ratio No.	3.96	3.64– 4.31	0.15	3.79%	4.04	3.62– 4.59	0.19	4.70%
Hydrocarbon	14.59%	12.28–17.09%	0.76%	5.21%				
Hcbn. f.p.	54.9°	53.8 –56.0°	0.54°	0.98%				
Sapon. cloud test	62.5°	61.8 –64.6°	0.52°	0.83%				

[a] Their samples 43, 44, 45, 49 omitted (see text).

wax were collected, as well as four special samples, described later. The samples of old comb wax were accepted only from producers who could certify that the comb foundation used was known to be 100% beeswax, without admixture of strengthening materials used by some manufacturers. When samples were received at the laboratory, they were melted in porcelain on a steam bath and filtered through paper. Two samples had to be boiled in water to allow subsequent filtration. After filtration the wax was melted, stirred, and poured into approximately 8 × 12″ aluminum foil pans to a depth of 4–7 mm and allowed to cool; the sheets obtained were cut into 5–7 mm squares. This procedure assured representative analytical samples. Preliminary hydrocarbon analyses of samples taken from various parts of a larger (500 g) block of wax indicated that special measures were needed to insure homogeneity of samples.

Analytical Methods

Hydrocarbon.—This was determined by a modification (14) of the chromatographic method of Bruening (13). The freezing point of the isolated hydrocarbon was determined as previously described (14).

Saponification No.—After a preliminary study to improve precision of this determination as applied to beeswax, the procedure below was adopted. The methods of Grodman (15), the AOAC (16), Warth (17), Paquot and Perron (18), and a method described by Rosenberg (19) were studied. Some gave erroneously high results because of indicator malfunctioning in virtually non-aqueous systems, others showed poor reproducibility. The method below was found to be substantially that of ASTM D 1387–59 (20), with some differences. It was found that Kimble[1] glass flasks were quite satisfactory for this use, but since they are no longer commercially available, Pyrex and Kimax were tried and found unsuitable. Corning alkali-resistant Glass #7280 was satisfactory.

To a sample of 1 g beeswax in a 300 ml alkali-resistant flask, add by pipet (90 seconds drain time) 40 ml ca 0.2*N* alcoholic KOH (95% ethanol) purified as described by the AOAC (16). Boil the flask, with alundum boiling chips, 3 hours under water-cooled reflux on a steam bath, with occasional shaking. Remove flask, add 50 ml 0.1000*N* HCl by pipet, and reheat to the boil. Add 1 ml of 1% phenolphthalein, and titrate on a hot plate with magnetic stirring to disappearance of pink color. The solution must be near boiling at the end point for reproducible results. Run a blank at the same time.

Acid No.—Dissolve 2 g wax in 100 ml neutral 95% ethanol, boil on a steam bath for 3 minutes, and titrate on a hot plate, with magnetic stirring, with 0.2*N* alcoholic KOH, using 1 ml 1% phenolphthalein. Blanks are necessary.

Ester No.—Difference between saponification and acid numbers.

Ratio No.—Ratio of ester number to acid number.

Color.—Estimate by visual comparison of the solid wax in ca 5 mm thickness, with Munsell color chips (21).

Melting Point.—The ASTM method for melting point of paraffin wax (D87–57 (22)) was used, with the exception that the tempera-

[1] Mention of trade names does not constitute endorsement by the Department over others of a similar nature not named.

ture was measured with an iron-constantan thermocouple and recorded, and the sample was stirred. The procedure was calibrated with three known compounds freezing in the temperature range of interest, using a thermometer calibrated at the National Bureau of Standards. The determination is actually that of freezing point under strictly defined conditions. The ASTM procedure is used by some beeswax processors.

Flash Point.—Cleveland open cup, ASTM D-92 (23).

Saponification Cloud Test.—Federal Specification C-B-191a (4).

Results and Discussions

Table 3 gives a summary of the analyses of 59 beeswax samples, with the range, standard deviation, and coefficient of variation. Similar data are given also in Table 3 for 56 samples of Western U. S. beeswax, from the work of Bisson, Vansell, and Dye (24). Four of their samples (No. 43, 44, 45, 49) have been omitted from our calculations because they varied grossly in their characteristics from the remaining 56. They were "comb honey scrapings wax," which would be much higher in propolis (resins) than comb or cappings wax.

Table 3 shows that the characteristic that varies least ($C_v = 0.55\%$) among our samples is the melting point. It might be expected that this value would be most useful in characterizing the quality of wax or demonstrating admixture with higher-melting microcrystalline wax. Grout (25) has shown the effect of adding a microcrystalline wax—m.p. (drop point) 77°C—to beeswax—m.p. (drop point) 64.4°. He found that up to 10% of the former raised the drop point only to 65.0–65.6°C (about 1°C).

Table 4. Melting point of beeswax-microcrystalline wax blends[a]

Microcrystalline Wax, %	Melting Point, °C	Increase, °C
0	63.78	
5	64.63	0.85
10	66.00	2.22
20	68.68	4.90
50	72.72	8.94
100	78.22	
Beeswax, average[b] $\pm 2s$	62.86–64.26°	
Beeswax, average $\pm 3s$	62.51–64.61°	
Beeswax, average $\pm 4s$	62.16–64.96°	

[a] ASTM Paraffin Wax Melting Point Determination.
[b] From Table 3.

Table 5. Flash points of waxes

Sample No.	Flash Point[a], °F
Cappings Waxes	
1	520
39	520
45	525
52	515
67	515
Old Comb Waxes	
8	490
17	510
23	510
54	505
73	520
Microcrystalline Wax[b]	570
70% Beeswax[c] 30% M.C. wax[b]	525
30% Beeswax 70% M.C. wax	540

[a] Cleveland open cup.
[b] Sample 18, Table 7.
[c] Sample 67.

Table 4 shows the melting points of several mixtures of beeswax and microcrystalline wax, determined by the same procedure used for the beeswax samples reported here. Also shown are intervals calculated from the data in Table 3—the $\pm 2s$ interval which should include 95.45% of beeswax samples, assuming a normal distribution; $\pm 3s$ which covers 99.73%; and $\pm 4s$ which includes 99.99% of samples. The 2.22° increase in melting point caused by addition of 10% of mineral wax is a bit greater than the $\pm 3s$ interval of 2.10°. It is less than the $\pm 3s$ interval (2.5°) calculated from the data of Bisson, *et al.* Thus, 10% addition of a sufficiently high-melting mineral wax might be detected by the melting point, using the apparatus described here.

The flash point has been suggested as a means of detecting mixtures of microcrystalline wax with beeswax. Little data on the flash point of beeswax appear in the literature. Stoeber in 1909 (26) gave the values for eleven samples of beeswax as

242–250°C (468–482°F), and that of most adulterants (at that time) as less than 200°C (392°F). Warth (27) gives the c.o.c. flash point of yellow beeswax as 468–482°F, and that of several microcrystalline waxes as 425–565°F. In Table 5 are given c.o.c. flash points of 5 samples of cappings wax and 5 samples of old comb wax, and those of a microcrystalline wax and two mixtures of the two. The beeswax values are higher than those reported by Stoeber and Warth, and addition of 30% of microcrystalline wax (flash point 570°F) raises that of the mixture only 10°F, so that it is still within the normal beeswax range. This determination is therefore not useful for this purpose.

The saponification cloud test is included in Federal Specifications for beeswax. It is required (4) that the temperature at which cloudiness begins be 65°C or below. Table 3 shows that the average temperature for the 59 samples is 62.5°C, with $s = 0.52°$. An interval of $\pm 4s$ gives 64.6° as the upper limit, within which only 1 in 16,667 genuine samples would not fall. The 65° limit set in the specifications is therefore quite liberal. Mixtures of 1, 2, 3, 4, 5, 10, and 20% microcrystalline wax and beeswax were tested. At 1%, the 61.8° cloud point of the beeswax was unchanged. The solution was slightly cloudy at the boiling point at 2 to 4%; at 5% and above, an insoluble layer at boiling was present; hence all except the 1% mixture failed the test. The 2 and 3% solutions were definitely cloudy when compared with controls.

The relative insensitivity of the common analytical "constants" of beeswax to admixture with inert waxes has been mentioned. By using the values for s for several determinations from Table 3, the minimum amount of inert material that could be detected through these analyses can be calculated. The sensitivity of this approach is limited by the extent of the interval representing normal variation in these "constants" in genuine beeswax. The fraction of genuine samples included within intervals of the mean $\pm 2s$, $\pm 3s$, and $\pm 4s$ (assuming a normal distribution) are shown in Table 6. Also the table shows the amounts of microcrystalline wax that could be added to a beeswax sample falling at the top of each interval without causing the value to fall below the stated lower limit. Thus, with interval limits of $\pm 3s$, one of every 370 samples of genuine beeswax would be expected to be rejected. Accepting this risk, at least 8% of mineral wax could be added to a beeswax with a saponification number at the top of the interval without bringing about rejection. To increase the sensitivity to 5.5% addition, one of each 20 samples of genuine wax would be rejected, an impractical situation. The situation is not actually as bad as this because samples showing high values for one constant do not necessarily also have high values for the others.

This objection does not apply to the hydrocarbon determination, since the material added is determined. Here it is necessary to consider the apparent hydrocarbon content of microcrystalline waxes, determined as for beeswax. Twenty samples of commercial microcrystalline waxes as shown in Table 7 were subjected to the determination as used for beeswax; the freezing point of the isolated hydrocarbon was also determined. These results are shown in Table 7. The average value obtained for hydrocarbon [2] by the chromatographic procedures is 84.74%, with $s = 1.21$. The values ranged from 79.63 to 91.27%. Since the addition of 1% of an average microcrystalline wax will add 0.85% of hydrocarbon, this will increase the

Table 6. Minimum amount of mineral wax detectable in beeswax by various determinations[a]

	Interval for Beeswax, in terms of s[b]		
	$\pm 2s$	$\pm 3s$	$\pm 4s$
Number of genuine samples in interval	19 of 20	369 of 370	16666 of 16667
Acid No.	14.0%	20.9%	27.9%
Sapon. No.	5.5%	8.25%	11.1%
Ester No.	6.0%	9.0%	12.1%

[a] Based on normal distribution.
[b] Data from Table 3.

[2] By "hydrocarbon" is meant the material passing through the alumina column in light petroleum ether solution.

Table 7. "Hydrocarbon" values of commercial micro-crystalline waxes

No.	M.P.[a], °C	Color[a]	"Hydrocarbon" %	"Hydrocarbon" f.p., °C
1	79.4–82.2	2 (ASTM)	91.27	67.1
2	—	Yellow[b]	84.29	78.6
3	71.1–76.7	White	84.65	70.8
4	—	Yellow[b]	87.67	76.2
5	81.1	Yellow 1¾ (NPA)	87.16	73.2
6	82.2–85.0	Amber 1½ (NPA)	80.13	78.7
7	73.9	White	88.13	73.1
8	76.7–79.4	White	87.42	72.9
9	73.9	Pale Yellow	84.96	72.8
10	82.2	Pale Yellow	81.95	78.6
11	79.4	White	85.11	71.2
12	78.3	Light Yellow	87.09	73.8
13	79.4–82.2	White	88.83	74.0
14	79.4	Amber[b]	79.63	74.4
15	82.2–85.0	White	86.83	74.8
16	76.7	Yellow	81.41	81.5
17	89.4	1¾ (NPA)	75.29	82.0
18	—	Light Amber[b]	87.61	73.8
19	—	Amber[b]	83.82	68.8
20	71.1–76.7	Amber	81.51	71.4
Mean			84.74	74.4
s			1.21	1.23
C_v			1.43%	1.65%

[a] As designated by manufacturer. [b] As estimated by authors.

hydrocarbon content of an average beeswax from 14.59 to 15.28%, producing a 4.73% change in hydrocarbon content.

It can also be calculated that at the $\pm 3s$ level $5.38\%\left(\frac{4.56}{.8474}\right)$ of an average microcrystalline wax could be added without raising the hydrocarbon content of a low-hydrocarbon beeswax above that of a high-hydrocarbon beeswax. Corresponding values for $\pm 2s$ and $\pm 4s$ are 3.58 and 7.16%. While this determination is somewhat more sensitive than those in Table 6, the freezing point of the hydrocarbon is still more sensitive. The average f.p. of the microcrystalline wax hydrocarbon fractions is 77.4°C, while that from beeswax is 54.9°C. The hydrocarbon isolated from mixtures shows freezing points between those of the two materials. The amount of increase in f.p. depends on the f.p. of the hydrocarbon of the particular microcrystalline wax present, and its amount. This is the reason for the variable per cent increase shown in Table 8 for the first four samples; the freezing points of the hydrocarbon from the microcrystalline waxes used were 82.0, 73.2, 67.1, and 68.8°C, respectively.

The intervals within which the hydrocarbon f.p. values for genuine domestic crude yellow beeswax can be expected to fall are

Table 8. Freezing point of hydrocarbon fraction of beeswax-microcrystalline wax blends

Composition % M.C. Wax[a]	F.P. of Hydrocarbon, °C	Increase[b], %
1	58.8	16
2	57.4	15
3	56.0	10
4	58.0	19
5	67.6	54
5	67.6	53
5	67.2	50
5	62.2	38
6	62.4	40
17	68.7	74
44	67.2	89
59	72.8	95

[a] Random pairings of 12 beeswaxes and 12 microcrystalline waxes from Tables 7 and 12.
[b] Expressed as per cent of difference beween f.p. of isolated hydrocarbon from specific waxes in each blend.

Table 9. Calculated ranges of freezing points of hydrocarbon fraction of U.S. yellow beeswax[a]

Interval	No. Included	Value, °C
$\pm 1s$	2 of 3	54.4–55.4
$\pm 2s$	19 of 20	53.8–56.0
$\pm 3s$	369 of 370	53.3–56.5
$\pm 4s$	16666 of 16667	52.8–57.0

[a] Assuming normal distribution.

shown in Table 9. The beeswax samples and the microcrystalline wax samples used to make these mixtures were selected and paired at random. It can be seen from Tables 8 and 9 that the presence of 5% of microcrystalline wax of the types described in Table 7 can be detected with a risk of rejecting only 1 genuine sample in 16,667.

Since both beeswax and microcrystalline wax vary in their hydrocarbon content as determined by alumina adsorption, calculation of the composition of a mixture from the hydrocarbon value found is at best an approximation. Table 10 gives the results of such analysis and calculation of composition of several mixtures. The samples of waxes used were all different, and selected and paired at random. The divergence between the known compositions and those found when calculations are based on the average values is of course wider than that calculated using the known hydrocarbon values for each member of the pair. The last two columns in Table 10 show the improved accuracy possible (s = 0.35 against 1.00) if the properties of the individual components are known.

The amount of microcrystalline wax in a mixture with domestic yellow beeswax may be estimated by the chromatographic determination of hydrocarbon content as previously described (14), and calculating:

$$\% \text{ Microcrystalline Wax} = 100\left(\frac{\% \text{ hcbn} - 14.59}{70.15}\right)$$

The standard deviation of the analysis, using these average constants, is 1% on the whole sample basis; if the hydrocarbon content of the components is known, this is reduced to 0.35%.

If the freezing point of the isolated hydrocarbon is 57°C or above (determined as previously described), the odds are 99,994 in 100,000 that it is not a genuine domestic yellow beeswax.

Data on Individual Samples.—Tables 11 and 12 show the physical and chemical data on the 63 individual beeswax samples. Samples 26–29, not included in the averages and calculations reported here, were produced at the Bee Culture Investigations Laboratory, Agricultural Research Service, Madison, Wisconsin. They were "scale" waxes, collected from caged colonies as follows:

	Rossman Hybrid Bees	*Starline Hybrid Bees*
Fed Sugar Sirup	No. 26	No. 28
Fed Clover Honey	No. 27	No. 29

None had access to pollen.

Summary

1. For 59 samples of crude yellow beeswax from the United States, the following average values were found, together with their standard deviations: melting point, 63.56°C (s = 0.35°); acid number, 18.33 (s = 0.64); saponification number, 90.94 (s = 1.35); ester number, 72.61 (s = 1.10); ratio number, 3.96 (s = 0.15); hydrocarbon, 14.59%

Table 10. Estimation of microcrystalline wax in yellow beeswax

Known Composition[a],	Composition Found			
	Using Averages		Using Individual Values	
%	%	Difference	%	Difference
0.93	1.78	+0.85	0.78	−0.15
2.12	1.92	+0.20	2.40	+0.28
3.14	3.35	+0.21	3.29	+0.15
4.02	3.76	−0.26	3.77	−0.25
4.79	4.09	−0.70	4.65	−0.14
4.54	4.58	+0.04	4.63	+0.09
4.79	4.68	−0.11	4.87	+0.08
4.88	3.43	−1.45	4.76	−0.12
6.07	6.81	+0.74	6.08	+0.01
17.49	16.39	−1.10	18.09	+0.60
43.60	44.89	−1.29	44.43	+0.83
59.23	61.30	+2.07	58.99	−0.24
Algebraic sum		−0.80		+1.14
s		1.00		0.35

[a] See footnote [a], Table 8.

Table 11. Physical properties of beeswax samples

No.	Origin	Type[a]	Color[b]	Melting Point, °C[c]
1	Columbia City, Ind.	C	5.0Y (7/6)	63.78
2	Galesburg, Mich.	OC	2.5Y (7/8)	63.45
3	Lowell, Mich.	OC	10.0YR (5/10)	63.58
4	St. Joseph, Mich.	OC	2.5Y (7/12)	63.08
5	Ft. Recovery, O.	C	2.5Y (7/10)	63.40
6	Wichita, Kan.	OC	10.0YR (4/4)	64.42
7	Pine City, Minn.	OC	2.5Y (7/8)	63.27
8	Shelbina, Mo.	OC	10.0YR (6/10)	63.10
9	Hamilton, Ill.	OC	10.0YR (4/6)	63.34
10	Streeter, N.D.	C	5.0Y (7/6)	63.50
15	Lake Leelanau, Mich.	OC	5.0Y (8/9)	63.83
16	Kent City, Mich.	OC	2.5Y (7/12)	63.63
17	Owosso, Mich.	OC	2.5Y (7/8)	63.27
20	Ortonville, Mich.	OC	2.5Y (6/10)	63.30
22	Taylor, Tex.	C	5.0Y (7/8)	63.83
23	Taylor, Tex.	OC	2.5Y (6.5/4)	63.50
24	Canton, O.	C	2.5Y (8/9)	63.36
25	Springfield, O.	C	5.0Y (7.5/8)	63.86
26	Madison, Wis.	S	White	62.92
27	Madison, Wis.	S	White	63.00
28	Madison, Wis.	S	White	63.18
29	Madison, Wis.	S	White	62.33
30	Chico, Calif.	OC	2.5Y (6/6)	63.34
31	Glenn, Calif.	C	3.5Y (7/8)	63.92
32	Orland, Calif.	OC	2.5Y (7/8)	63.88
33	Porterville, Calif.	C	5.0Y (6.7/4)	63.36
34	Modesto, Calif.	C	5.0Y (7/8)	63.36
35	Palo Cedro, Calif.	C	4.0Y (7/6)	63.51
36	Modesto, Calif.	C	2.5Y (7/8)	63.51
37	Tulare, Calif.	C	2.5Y (6/4)	63.84
38	Livingston, Calif.	OC	5.0Y (8/5)	64.27
39	Cumberland, Md.	C	5.0Y (8/8)	63.50
40	Red Springs, N.C.	C	5.0Y (8/6)	63.56
41	Rural Hall, N.C.	OC	2.5Y (6.5/8)	63.39
42	Glenarm, Md.	C	5.0Y (7/6)	63.78
43	S. Deerfield, Mass.	C	5.0Y (7.5/6)	63.92
44	Rural Hall, N.C.	C	5.0Y (7.5/8)	63.92
45	Lynchburg, Va.	C	5.0Y (8/5)	63.56
47	S. Deerfield, Mass.	OC	2.5Y (7.2/8)	63.43
48	Seville, O.	C	5.0Y (7.5/10)	64.21
49	Akron, O.	C	5.0Y (7.5/4)	64.24
50	Wellston, O.	C	5.0Y (7.5/7)	64.12
51	Elyria, O.	C	5.0Y (7.5/7)	63.92
52	Wapato, Wash.	C	5.0Y (7.5/6)	63.53
53	Livingston, Mont.	C	2.5Y (7.5/10)	63.50
54	Livingston, Mont.	OC	2.5Y (6/10)	63.30
55	Boise, Idaho	C	5.0Y (8/9)	63.86
57	Boise, Idaho	C	5.0Y (7.5/6)	63.93
58	Boise, Idaho	OC	2.5Y (7/6)	63.43
59	Durango, Colo.	C	5.0Y (7/12)	64.40
61	Wolf Point, Mont.	C	5.0Y (7.5/7)	63.61
62	Wolf Point, Mont.	OC	2.5Y (6/6)	63.03
63	Meeker, Colo.	C	5.0Y (11/7.5)	62.68
64	Meeker, Colo.	OC	10.0YR (7/12)	63.13
65	Marshall, Mich.	C	5.0Y (7.5/12)	63.41

(Continued)

Table 11 (*Continued*)

No.	Origin	Type[a]	Color[b]	Melting Point, °C[c]
66	Marshall, Mich.	C	5.0Y (8/9)	63.60
67	Cannon Falls, Minn.	C	5.0Y (8/7)	63.13
68	Paris, Tex.	C	7.5Y (8/8)	63.69
69	Monte Vista, Colo.	OC	10.0YR (4/6)	63.05
70	San Angelo, Tex.	OC	2.5Y (6/7)	62.85
71	Jeanerette, La.	C	5.0Y (8/9)	63.31
72	Tahlequah, Okla.	OC	10.0Y (5/8)	63.66
73	Garland, Tex.	OC	2.5Y (6/8)	63.60

[a] C=capping; OC=old comb; S=scales.
[b] Munsell Notation (21).
[c] Old comb waxes: Average 63.44°, Range 62.85–64.42°.
Capping waxes: Average 63.66°, Range 62.68–64.40°.
All waxes (except 26–29): Average 63.56°; s=0.35°; C_v=0.55%.
Average of 26–29: 62.86°.

Table 12. Chemical Values of Beeswax Samples

No.	Acid No.	Sapon. No.	Ester No.	Ratio No.	Sapon. Cloud, °C	Hydrocarbon, %	Hcbn. f.p., °C
1	19.06	94.29	75.23	3.95	62.6	14.27	55.0
2	18.12	91.42	73.30	4.04	62.2	14.80	56.0
3	20.04	94.39	74.35	3.71	62.4	12.28	56.0
4	17.54	88.82	71.28	4.06	62.0	17.09	53.8
5	19.77	92.39	72.62	3.67	62.4	15.10	54.7
6	17.78	90.64	72.86	4.10	62.2	12.92	55.9
7	18.28	92.13	73.85	4.04	62.2	15.56	54.4
8	18.74	92.93	74.19	3.96	62.8	15.38	54.3
9	19.19	90.68	71.49	3.72	62.6	15.15	54.2
10	19.20	93.66	74.46	3.88	62.2	13.98	55.3
15	17.38	90.50	73.12	4.21	63.0	14.90	55.7
16	17.80	90.90	73.10	4.11	62.6	15.91	54.4
17	18.68	90.84	72.16	3.86	62.8	14.52	54.6
20	18.08	88.96	70.88	3.92	62.8	15.63	54.7
22	18.57	92.88	74.31	4.00	61.8	13.83	55.6
23	19.33	90.88	71.55	3.70	62.8	13.66	55.5
24	20.12	93.32	73.20	3.64	62.6	13.98	55.5
25	18.54	92.74	74.20	4.00	62.0	14.47	55.3
30	18.05	90.11	72.06	3.99	62.8	14.65	55.8
31	16.68	88.62	71.94	4.31	62.8	14.73	54.6
32	17.33	89.08	71.75	4.14	62.8	15.18	54.6
33	18.03	90.64	72.61	4.03	61.8	14.49	55.1
34	17.96	90.76	72.80	4.05	62.0	14.56	54.8
35	17.96	90.47	72.51	4.04	62.2	15.03	55.4
36	17.53	89.08	71.55	4.08	62.6	15.40	54.6
37	17.68	88.93	71.25	4.03	62.6	14.68	54.4
38	17.60	90.48	72.88	4.14	62.4	13.33	55.1
39	17.87	90.33	72.46	4.05	62.2	14.41	54.7
40	17.49	89.37	71.88	4.11	61.8	14.19	54.7
41	18.09	89.99	71.90	3.97	62.0	15.21	54.7
42	18.02	89.84	71.82	3.98	62.4	14.75	55.2
43	17.60	90.50	72.90	4.14	64.6	14.41	55.1
44	18.25	90.86	72.61	3.98	62.2	14.04	55.5
45	18.65	90.23	71.58	3.84	62.0	13.68	55.2
47	17.45	88.63	71.18	4.08	61.8	15.31	55.1

Table 12 (*Continued*)

No.	Acid No.	Sapon. No.	Ester No.	Ratio No.	Sapon. Cloud, °C	Hydrocarbon, %	Hcbn. f.p., °C
48	17.30	90.21	72.91	4.21	63.8	14.32	55.0
49	17.75	91.15	73.40	4.13	62.6	13.64	55.9
50	17.87	89.45	71.58	4.00	62.4	14.77	55.5
51	18.13	89.41	71.28	3.93	63.4	14.30	55.2
52	18.07	91.25	73.18	4.05	62.2	14.62	55.4
53	18.37	91.62	73.25	3.99	62.2	14.10	55.4
54	18.73	90.26	70.99	3.68	63.0	15.47	54.6
55	18.73	91.05	72.32	3.86	63.6	13.73	55.2
57	18.07	90.61	72.54	4.01	62.6	13.65	55.0
58	18.84	90.63	71.79	3.81	62.6	15.02	54.6
59	18.42	93.74	75.32	4.09	62.8	14.01	55.2
61	18.67	92.26	73.59	3.94	62.0	14.92	54.0
62	19.06	90.97	71.91	3.77	62.0	15.47	54.4
63	18.67	92.67	74.00	3.96	61.8	13.75	54.4
64	18.16	92.69	74.53	4.10	62.8	14.87	54.1
65	18.68	91.13	72.45	3.88	62.2	15.28	54.4
66	18.99	90.75	71.76	3.78	62.4	14.43	54.8
67	18.87	90.82	71.95	3.81	62.2	13.96	55.5
68	18.46	89.38	70.92	3.84	62.8	14.57	55.1
69	17.89	90.45	72.56	4.06	62.2	15.49	53.8
70	18.61	89.43	70.82	3.80	61.8	15.13	54.3
71	19.05	92.46	71.64	3.76	62.2	14.23	55.8
72	18.04	90.69	72.65	4.03	62.4	14.44	55.4
73	18.67	91.55	72.88	3.90	62.6	14.57	55.2
			Special Samples				
26	20.63	92.36	71.73	3.48	61.0	12.55	55.1
27	21.06	93.89	72.83	3.46	60.4	12.54	55.4
28	20.45	94.04	73.59	3.60	60.2	11.27	55.4
29	19.69	93.55	73.86	3.75	60.4	12.30	55.2
Av.	20.46	93.46	73.00	3.57	60.5	12.16	55.3
			25 Old Comb Waxes				
Av.	18.33	90.72	72.39	3.95	62.5	14.88	54.8
Range	17.30–20.04	88.63–94.39	70.88–75.32	3.70–4.21	61.8–63.8	12.28–17.09	53.8–56.0
			34 Cappings Waxes				
Av.	18.33	91.08	72.75	3.97	62.5	14.36	55.1
Range	16.68–20.12	88.62–94.29	70.82–75.23	3.64–4.31	61.8–64.6	13.64–15.40	54.4–55.9
			59 Cappings and Old Comb Waxes				
Av.	18.33	90.94	72.61	3.96	62.5	14.59	54.9
s	0.35	1.35	1.10	0.15	0.52	0.76	0.52
C_v	0.55%	1.49%	1.51%	3.79%	0.83%	5.21%	0.83%

($s = 0.76\%$); hydrocarbon freezing point, 54.9° ($s = 0.54°$); saponification cloud test, 62.5° ($s = 0.52°$).

2. The presence of 5% or more microcrystalline wax can be detected with certainty by the freezing point of the hydro-

carbon isolated in the chromatographic determination of hydrocarbon.

3. The microcrystalline wax content of a mixture with yellow beeswax may be estimated by determining the hydrocarbon content of the mixture.

4. The 65°C upper limit of the saponification cloud test is a reasonable value. This test will detect 2% of microcrystalline wax in domestic crude yellow beeswax.

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